Properties of As-grown, Chemically Treated and Thermally Oxidized Surfaces of AlGaN/GaN Heterostructure

S. Ootomo, S. Oyama, T. Hashizume and H. Hasegawa
Research Center for Interface Quantum Electronics,
and Graduate School of Electronics and Information Engineering, Hokkaido University,
North 13 West 8, Sapporo, 060-8628, Japan.
E-mail address: ootomo@rciqe.hokudai.ac.jp

1. Introduction

The AlGaN/GaN heterostructure has recently turned out to be an extremely important starting structure for fabrication of high-voltage/high-power HEMTs operating at microwave/millimeter-wave frequencies. Impressive frequency/power performances of devices as well as basic electrical properties of 2DEG at AlGaN/GaN heterointerface have been reported by many workers, showing high potentials of these devices. However, properties of the surface of the AlGaN/GaN heterostructure are not well understood in spite of the fact that any device processing step is initiated on this surface. Optimized control of the starting surface is an essentially important step for formation of stable Schottky gates and good ohmic contacts as well as for successful surface passivation.

In this paper, properties of as-grown, chemically treated and thermally oxidized surfaces of the AlGaN/GaN heterostructure are systematically studied by x-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), surface Raman spectroscopy, Hall-effect, capacitance-voltage and magneto-transport measurements.

2. Experimental

The structure of $Al_{0.25}Ga_{0.75}N$/GaN heterostructure samples used in this study is shown in **Fig. 1**. The structure was grown by MOVPE on (0001) sapphire substrate. As a first step, properties of the air-exposed surface of the as-grown wafer were investigated. Then, in order to control the natural oxide layer on the air-exposed AlGaN surface, the effect of the following three kinds solutions for wet chemical treatments were investigated: (i) a KOH solution at room temperature (RT) for 1 min, (ii) an NH_4OH solution at 50°C for 15 min and (iii) an HF solution at RT for 15 min. Finally, in order further to compare surface chemistry as well as to explore possibilities of oxide surface passivation, AlGaN surfaces were intentionally oxidized thermally at 900°C for 30 min in O_2.

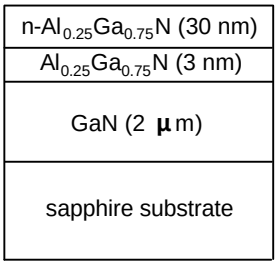

Fig. 1 sample structure.

3. Results and Discussion

Basic electrical properties of heterointerface

The Hall mobility and sheet carrier concentration of 2DEG formed at the AlGaN/GaN heterointerfaces were 1200 cm^2/Vs and 1.6×10^{13} cm^{-2} at RT, respectively. These are the typical values comparable to those reported in the literature. The Shubnikov de Hass oscillations were clearly observed above 4 T, indicating existence of 2DEG at the heterointerface. These results conformed that our AlGaN/GaN heterostructure was of device quality with a high-quality AlGaN toplayer, and worth while for further investigation.

Air-exposed surface of as-grown heterostructure

From the XPS measurements, a high-intensity O1s peak was detected on the air-exposed AlGaN surface, being accompanied with broadening of Al, Ga, N and O core-level spectra. **Figure 2 (a)** shows the angle-resolved XPS spectra of the Al2*p* level obtained from such a surface. All spectra had shoulders on the higher binding energy side. In particular, the spectrum obtained at an escape angle of 10° exhibited a peak position very close to that of the Al-O bond in the Al_2O_3 phase. A similar feature appeared in the Ga3*d* core-level. Judging from the energy positions of the Al-O bond in Al2*p* and the Ga-O bond in Ga3*d*, the air-exposed surface was covered with a natural oxide layer consisting of the Al_2O_3 and Ga_2O_3 components.

The XPS integrated intensity ratios of O1*s* to N1*s* (O1*s*/N1*s*), Al2*p* to N1*s* (Al2*p*/N1*s*) and Ga3*d* to N1*s* (Ga3*d*/N1*s*) are summarized in **Fig. 2 (b)** as a function of the escape angle. The O and Al components rapidly increased as the escape angle was decreased, indicating that the Al_2O_3 component is dominant in the natural oxide layer. From a detailed analysis, the thickness of the natural oxide layer was estimated to be 0.4-0.6 nm with the Al_2O_3 to Ga_2O_3 composition ratio of 2.3-4.0.

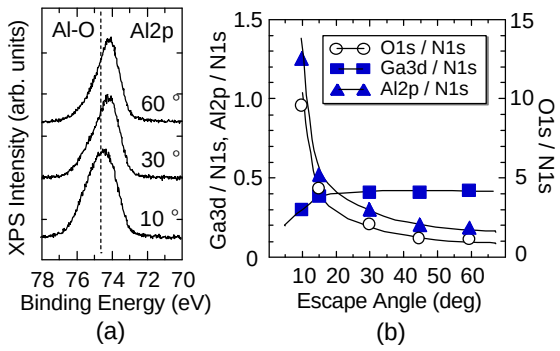

Fig. 2 (a) Angle-resolved XPS spectra and (b) integrated intensity ratios on air-exposed surface.

Effects of chemical treatments

In order to remove the natural oxide layer from the air-exposed surface, three kinds of chemical treatments mentioned previously were applied. As a result, all the treatments significantly reduced the O1*s* peak height on the AlGaN surface as shown in **Fig. 3**. However, surface chemistry was very different. For the KOH- and HF-treated surfaces, surface stoichiometry was disturbed with Ga-rich and Al-rich features as well as appearance of K- and F- related residual XPS peaks, respectively. Such surfaces seem unfavorable due to possibilities of causing interfacial instabilities and unwanted reactions on deposition of metal contacts and insulating films during device processing.

On the other hand, the spectra after the NH_4OH treatment had no noticeable impurity peaks. They had almost stoichiometric Al-N and Ga-N bonding components from the following reasons. Namely, as shown in **Fig. 4 (a)**, the Al2*p* peak became shoulderless with a significant peak energy shift toward a lower binding energy after the NH_4OH treatment. This peak position is very close to that of the Al-N bond. The energy peak of the Ga3*d* spectra also moved to the position corresponding to the Ga-N bond. Furthermore, the data of integrated intensity ratios shown in **Fig. 4 (b)** indicated that this AlGaN surface has a spatially uniform composition with a remarkable reduction of the O1*s* intensity. The Al composition was estimated to be 23% with reference to the Ga3*d*/N1*s* value in the reference GaN sample grown on sapphire by MOVPE, indicating that the NH_4OH-treated AlGaN surface has a composition close to the stoichiometric value (25%). **Figure 5** shows the Raman spectra obtained from the AlGaN/GaN heterostructure samples exposed to air and treated in an NH_4OH solution. The NH_4OH treatment enhanced the intensity of the E_2 peak originating from the AlGaN lattice, as shown in the inset, indicating that considerable improvement of the crystalline quality of the surface takes place by the NH_4OH treatment.

All these results indicate that the NH_4OH treatment is very effective in realizing an oxide-free and well-ordered AlGaN surface.

Thermally oxidized AlGaN surface

Properties of the surface obtained after intentional thermal oxidation of the NH_4OH-treated surface was further investigated. No noticeable N-related peaks were detected in both the XPS and AES spectra from such a surface. The XPS intensity ratios of the Al2*p*/O1*s*, Ga3*d*/O1*s* and Al2*p*/Ga3*d* are plotted in **Fig. 6** as a function of the escape angle. These ratios are almost constant in the depth direction at least down to escape depths of photoelectrons, indicating that the surface was covered with a much thicker oxide layer with spatially uniform composition distribution of Ga-oxide and Al-oxide components. The Al2*p* and Ga3*d* levels also have constant peak positions corresponding to those of Al_2O_3 and Ga_2O_3, respectively. The composition of Al_2O_3 in the oxide layer was estimated to be 0.22, corresponding to the Al content in the AlGaN bulk. This feature is very different from the surface natural oxide and provides a great hope for surface passivation by thermal oxidation.

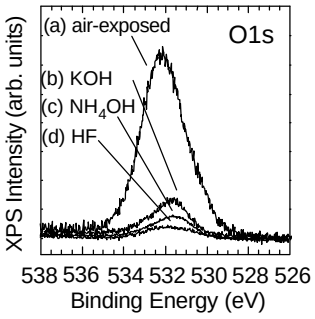

Fig. 3 O1*s* spectra of AlGaN surfaces

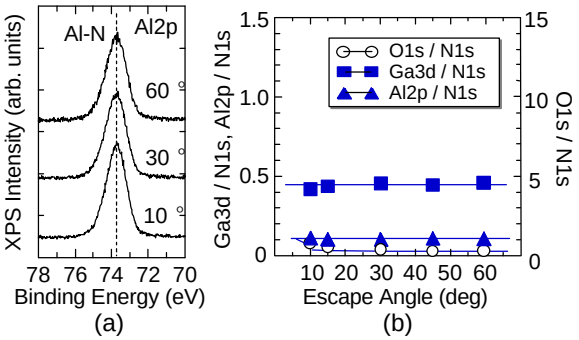

Fig. 4 (a) Angle-resolved XPS spectra and (b) the integrated intensity ratios from the NH $_4$OH-treaed surfaces.

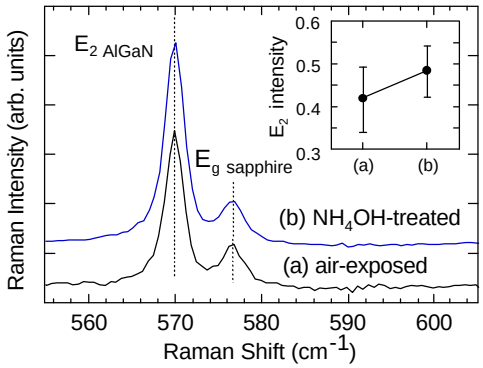

Fig. 5 The Raman spectra obtained from (a) exposed surface and (b) NH_4OH-treated surface.

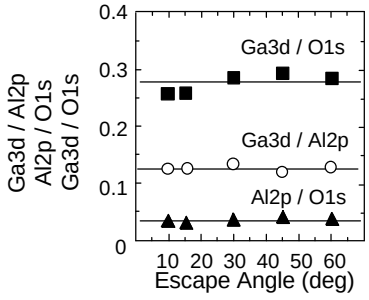

Fig. 6 The integrated intensity ratios after thermal oxidation for 30 min at 900 °C.